PATENT SPECIFICATION

DRAWINGS ATTACHED

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1,112,023

1,112,023

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Int. Cl.:—**B 29 c 13/02**

COMPLETE SPECIFICATION

Process for the Production of Gasketed Container Closures

We, W. R. GRACE & Co., a Corporation organized and existing under the laws of the State of Connecticut, United States of America, of 62 Whittemore Avenue, Cambridge 40, Massachusetts, United States of America, do hereby declare the invention for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

THIS INVENTION relates to a process for the production of gasketed container closures.

To retain the contents of a container within the container, (usually fabricated from glass, plastic, metal or a laminate), a closure seal is required between the pouring rim of the container and the retaining closure. With some packs, that is a container and its contents, the closures seal is required to hermetically seal the contents from bacterial contamination. Other container closures are required to exhibit resealing features and some are required to withstand processing at high temperatures and pressures.

The invention is of particular interest in connection with seals in closures of the crown type. It has been proposed, and is a practice, to line crown shells by flowing a liquid or semi-liquid plastisol into a crown and then either spinning the crown to distribute the composition before fluxing, or moulding with a hot mould to flux or partially flux the composition while shaping the gasket. In both cases the plastisol is subsequently gelled i.e. liquefied, either in an oven or by further moulding.

It has also been proposed to line crown shells by introducing pre-formed inserts, e.g. pre-moulded polyethylene liners. The closure insert may be introduced into the closure, for example by any of the means listed below. These are illustrated in the accompanying Drawings, in which Figures 1—7 are cross-sections taken across different types of circular closures.

1. A uniform overall disc of resilient sealing material is inserted into the closure and retained by an adhesive or by restrictions formed in the closure. Such discs are cut or stamped from sheets, strips, or coils of sealing material. These methods are illustrated in Fig. 1 and Fig. 2 wherein the disc of resilient sealing material is represented by 1, the container closure by 2 and the adhesive (Fig. 1 only) by 3.

2. A contoured disc of resilient material is inserted into the closure and retained by an adhesive or by restrictions formed in the closure. Such inserts are usually injection moulded or compression moulded from thermoplastic materials or non-thermoplastic materials, containing resinous binders. These methods are illustrated in Fig. 3 and Fig. 4 wherein the contoured disc of resilient material is represented by 4, the container closure by 2 and the adhesive (Fig. 3 only) by 3.

3. An annular or overall resilient material is transferred from a coated sheet to the closure by the application of pressure. This is illustrated in Fig. 5. The transfer die 7 and/or metal plate 6 may be heated to assist transfer of the material 8 and an easy release coating may be used between the coating of closure material 8 and the backing 9. This technique is known in the prior art as "hot foil printing".

4. A cut or moulded ring of resilient sealing material, made from vulcanized rubber or compounded PVC, which is retained by the closure and the compressibility of the seal is placed in the closure. An adhesive may be

used to retain the ring in the closure. This is illustrated in Figs 6 and 7 wherein 10 represents the sealing ring and 2 the closure.

When a crown shell is used for capping a bottle or other container the gasket is required to conform to possible irregularities in the finish of the lip of the container so as to provide a good seal between the closing face of the metal crown shell and the contents of the container, which frequently contain dissolved gases under pressure. Usually the crown gasket has also the further function of preventing contact of the contents of the container with the metal of lacquered metal of the inner face of the crown shell. Both of these functions require the use of gasketing materials that are essentially impermable to the contents of the pack, but a much thinner layer is needed to protect the crown shell from contact with the contents than to effect the seal.

According to one aspect of the invention, a process for the production of gasketed container closures includes introducing a preformed insert of a solid thermoplastic resilient material into a container closure which is warmed sufficiently to cause adequate adhesion of the insert to the closure, and moulding the insert to the required shape under pressure, with a cold moulding member, if necessary after further heating the closure.

This process has a number of advantages. In the first place it provides a simple method of forming a sealing gasket with a relatively thick sealing portion and a thinner central protective panel, e.g. by use of a suitable moulding member, a crown seal in which the sealing composition is contoured to give a relatively thick annular sealing portion and a thinner central panel which protects the metal or lacquer of the crown shell can be obtained, thus enabling less of the thermoplastic composition to be used than if the gasket were of the same thickness overall. Moreover, waste thermoplastic material from the blanking or shaping process by which the inserts are made can be easily reworked into sheet or rod form, and cut to provide further inserts for use according to the process of the invention. Existing equipment for inserting cork discs or laminated paper wads can readily be adapted to the new process, and the use of a cold moulding member is found to give the gasket a high quality finish.

By the term "cold moulding member" whenever used in this specification, we mean a moulding member which is not heated except as a result of its contact with the container closure insert in the moulding operation. It will be understood that although the moulding member is not heated, and indeed will usually be positively cooled, its temperature will necessarily be raised to some degree by conduction from the heated insert. It is essential however that the moulding member remains "cold" in relation to the temperature of the insert.

It is also found that the new process does not suffer from difficulties caused by the trapping of air between the gasket material and the metal of the closure, such as have characterised some known processes. A high degree of uniformity in placement and weight of material can be obtained over long runs with very little supervision. Clear liners, for use over lithographed or otherwise decorated or incribed closures, can be formed if desired. Finally, the process does not require the use of hot ovens or complex metering equipment, or a precise viscosity control.

The thermoplastic resilient material to be used should possess sufficient elasticity to provide a good seal over the range of temperatures to which the sealed container may be exposed, adhere well to the lacquer (or metal) of the metal closure, when heated flow sufficiently to be moulded under pressure, and be sufficiently rigid at room temperature to enable the inserts to be handled with automatic equipment. For food closures it should naturally be non-toxic. Suitable materials are discussed hereinafter.

It is possible by the process of the invention to manufacture crowns having excellent sealing performance at low film weights, for example about 150 ms. of the composition in a standard crown shell, e.g. from thermoplastic resilient materials described in our British Specification No. 1,092,161.

The pre-formed inserts are preferably, though not necessarily, circular, and may be obtained for example either by cutting or stamping out of cast, calendered, pressed or extruded sheet, or by cutting from rod, or by injection moulding. They will usually be considerably smaller in diameter than the closure, and of course thicker than is required for the central protective panel in the finished article.

The process of this invention can be used to provide sealing gaskets not only in crown shells of the standard or short skirt type, but also in other types of closures such, for example, as pilfer-proof caps, lug caps, plastic caps, rolled-on caps, and crimped-on caps.

The process may be carried out using a modified standard cork disc inserting equipment. The closure and the insert may be heated by direct conduction, convection or radiant heating or ultrasonic heating. The insert may also be heated by a radio frequency technique or dielectric heating, and the metal closure by induction heating. When two heating stages are used, the same or different methods may be used in each.

The moulding step can conveniently be carried out as a continuous operation, for example using a series of plungers set in the periphery of a wheel. If necessary the mould-

ing plunger or plungers should be cooled after each moulding operation. However, the actual method used for the cold moulding is not critical, and many of the moulding devices known to the art can be used, provided they can be kept cold during the actual moulding operation. Likewise the configuration of the moulding parts of the plunger can be varied to give any desired configuration to the liner.

A method of carrying out the process of the invention is illustrated in Figures 8—13 of the accompanying drawings, which represent diagrammatically successive stages in the formation of the finished article.

Referring now to the drawings, Figure 8 shows the first stage in the process, in which a lacquered metal crown shell 11 is heated on a heating plate 12 provided with an electrical resistance heater 13; other methods of heating the plate can, however, be used if desired. In this stage the shell is heated to a temperature at which a disc of the thermoplastic resilient composition can be caused to adhere to the lacquer surface at least sufficiently firmly to hold it in position during subsequent handling; generally a temperature of about 40°—120° C. will suffice, but this will of course depend largely on the particular material used.

The next stage, shown in Figure 9, involves the insertion of the disc centrally in the heated shell. This can be done automatically using a tubular feeding member 14 adapted to fit more or less closely within the inverted shell, and provided with a plunger 15 by means of which a disc 16 can be pressed against the heated shell 11 sufficiently firmly to cause the required degree of adhesion. After each disc has been placed in position the shell is removed and replaced by another by a conventional mechanism (not shown), and another disc is fed into the tube below the plunger from a pile 17. After being moved from below the feeding device the shell, now containing the adherent disc as shown in Figure 10, passes (Figure 11) to a further heating point 18, where by means of a heater 19, shown as a resistance heater, it is brought to a temperature at which the disc becomes capable of sufficient flow for the moulding operation. This temperature again will depend on the nature of the thermoplastic composition of which the disc consists; usually it will be above 130° C., e.g. 180°—250° C. It will be understood that other forms of heater may be used at this point, e.g. a radiant infrared heater.

The heated shell now passes to a moulding position as shown in Figure 12. Here it is placed in a recessed holder 20 supported on heat-insulating material 21 to prevent undue loss of heat, under a moulding member 22 having its lower face 23 shaped to form the annular sealing gasket and the much thinner central protective panel required. The moulding member is provided with cooling means, exemplified in the Figure by inlet and outlet pipes 24, 25 for a cooling fluid, e.g. cold water. After the moulding operation the lined shell, in which the lining now has the configuration shown in Figure 13, is removed from the moulding position in any conventional way, and is ready for use. It is frequently advantageous to have the base of the recess curved to correspond to the curvature of the shell.

Any conventional or known means may be provided for moving the shells from one station to another. For example they may be carried on a horizontal wheel through the heating stations and under the feeding member 14, and thence passed by gravity feed down a chute to the moulding position.

In our Specification No. 33863/67, (Serial No 1112025) we have described and claimed a method of forming sealing gaskets in container closures, which comprises placing within the closure a layer of a composition containing a thermoplastic resilient material comprising

(a) a copolymer of an α-mono-olefine with another α-olefinically unsaturated monomer, or

(b) a mixture of a poly-α-mono-olefine with a polymer of one or more α-olefinically unsaturated monomers, said polymer not being a poly-α-mono-olefine, or

(c) a mixture of polyisobutylene with another poly-α-mono-olefine, and causing the layer to adhere to the closure by the action of heat, pressure or an adhesive. The term "α-mono-olefine" means an unsubstituted α-mono-olefine and the term "poly-α-mono-olefine" means a homopolymer of an α-mono-olefine. The term "α-olefinically unsaturated monomer" includes any monomer containing at least one ethylenic double bond in the α-position. Thus, besides α-mono-olefines, such compounds as vinyl acetate, acrylic acid, acrylic acid esters and isoprene fall within the definition.

Copolymer (a) referred to above is a copolymer of an α-mono-olefine, e.g. ethylene, with another α-olefinically unsaturated monomer, which may be another α-mono-olefine such as propylene, or a non-α-mono-olefine such as vinyl acetate or acrylic acid. Copolymer (a) may be formed from more than two monomers. For instance, it may be a copolymer of one α-mono-olefine with another and with an α-olefinically unsaturated monomer, e.g. a rubbery terpolymer comprising ethylene and propylene as the major ingredient, and a third α-olefine as a minor ingredient.

In a particular embodiment of the invention of Specification No. 33863/67, the thermoplastic material comprises a mixture of copolymer (a) and a poly-α-mono-olefine, preferably polyethylene and most preferably low

density polyethylene. Among the mixtures which may be used in the method of the invention, the following are preferred types:

(a) a copolymer or mixture of copolymers of an α-mono-olefine and a vinyl or acrylic ester in admixture with a polymer of one or more α-mono-olefines.

(b) a mixture of a poly α-mono-olefine with a rubbery copolymer of ethylene and propylene, or with a rubbery terpolymer comprising ethylene and propylene as the major ingredients and a third α-olefine as a minor ingredient.

(c) a mixture of a poly α-mono-olefine and a chlorosulphonated polyethylene.

(d) a mixture of poly-α-mono-olefine and a polyisobutylene or a mixture of a poly-α-mono-olefine and a copolymer of isobutylene with a minor proportion of isoprene.

The poly-α-mono-olefine referred to is preferably polyethylene, especially low density polyethylene.

We have found the following specific materials to be particularly suitable:

(1) Materials described in our Specification No. 1,092,161.
(2) Copolymers of ethylene and vinyl acetate.
(3) Mixtures of copolymers of ethylene and vinyl acetate with polyethylene.
(4) Copolymers of ethylene and acrylic acid including esters thereof.
(5) Mixtures of copolymers of ethylene and acrylic acid, including esters thereof, with polyethylene,
(6) Plasticized polyvinyl chloride.
(7) Mixtures of copolymers of ethylene and propylene with polyethylene.
(8) Mixtures of polyethylene with chlorosulphonated polyethylene.
(9) Mixtures of polyethylene with butyl rubber.
(10) Mixtures of polyethylene with polyisobutylene.
(11) Mixtures of ethylene/vinyl acetate copolymers with ethylene/propylene copolymers.
12) Mixtures of any of the compositions (4), (5), (7), (8), (9), and (10) with ethylene/vinyl acetate copolymers, and in particular mixtures of polyethylene, butyl rubber, and ethylene/vinyl acetate copolymers (described and claimed in our Specification No. 33862/67). (Serial No 1112024).
(13) Polyethylene.
(14) Mixtures of polyethylenes of different densities, molecular weights, or molecular weight distributions.

The ethylene/vinyl acetate polymers will normally contain 5—50%, preferably 12—40%, of vinyl acetate.

The process of the invention can be used to provide sealing gaskets in many types of closures, for example in crown shells of the standard or short skirt type, pilfer-proof caps, lug caps, plastic caps, rolled-on caps, and crimped-on caps. Some of the materials have special utility with rolled-on and pilfer-proof caps.

The advantages of materials (2)—(5) and (7)—(12) are described in the specification of Application No. 33863/67 (Serial No 1112025). Blends of polyethylene, butyl rubber and a copolymer of ethylene and vinyl acetate, when formed into crown or pilfer-proof shells by the cold-moulding process of the invention, give sealing gaskets which have excellent sealing properties (including retention of pressure in the pack) for long periods at elevated temperatures, which may be made in quite a wide choice of colours without significant loss in sealant properties, which impart a generally lower level of taste contamination than plasticized polyvinyl chloride, which has a greater resistance to physical abuse than polyethylene, and which do not require the use of a special adhesive as does polyethylene.

The thermoplastic resilient material may contain the various components of the copolymers or polymer mixtures in any proportions compatible with the properties desired in the sealing composition; such proportions, if in any case they are not already known, are easily determined by routine experiment.

Fillers, pigments, colouring materials, slip agents, blowing agents, cork particles, resins, rust inhibitors, and other conventional additives may be added to any of the materials (2) to (7) as required to give particular effects. When a blowing agent is present, it is usually found that the thin protective panel section of the liner is not, or not greatly, expanded, while the raised annular sealing section has a typical cellular structure. Blowing agents and other additives can be of the conventional kinds. Thus, typical pigments that may be used are titanium dioxide and carbon black, while as blowing agents there may be used known azo compounds, e.g. azobiscarbonamide. Examples of fillers which may be used are chalk (and other forms of calcium carbonate), barytes and clays, Slip agents such as silicones and waxes may be used. Suitable resins include rosin and rosin esters and terpene resins. Rust inhibitors include for example sodium benzoate, which may be present for example in amount 1—3% of the weight of the composition.

The invention is illustrated by the following Examples. In Examples 1—5 a procedure as described above by reference to Figures 8 to 13 of the drawing was employed. "Parts" are by weight.

EXAMPLE 1

The following materials were mixed together in a heated vessel at 150° C. with stirring

for 30 minutes, being added in the order given:

	parts
Abietic acid ester (Staybelite Ester 5)	30
Microcrystalline Wax	30
Ethylene/vinyl acetate copolymer (27—29% vinyl acetate)	62
Whiting	5
Titanium Dioxide	5

The air was removed from the system, and the molten composition poured into a flat cold mould, and while still warm compressed in a hydraulic press at 210 kg./cm.^{2} to give a sheet 0.14 cm thick.

Discs of diameter 1.1 cm. were cut from the sheet. The off-cut material was re-melted and pressed again using the same technique into another sheet of the same thickness, from which further discs were cut.

Crown shells internally coated with a vinyl lacquer were heated on the heated plate (Figure 8) whose surface temperature was approximately 60° C. The discs were inserted centrally into the crowns, while substantially retaining their shape, as shown in Figure 9. At this stage the shells containing the discs could be handled without the discs being dislodged, and if required could be stored for further processing at a later date. At the next heating station (Figure 11) each shell rested for 25 seconds on a heating plate having a surface temperature of approximately 230° C. so rendering the material of the disc semi-fluid. The shell was then passed to the moulding station (Figure 12) where the gasket was cold-moulded into the shape shown in Figure 13.

The weight of the liners so formed was 152±4 mg. The crowns were found to have excellent sealing characteristics when subjected to the following high carbonation retention tests. Glass bottles packed at three volume and five volume carbonation were stored at 38° C. for one month, and were then examined. No loss of pressure had taken place.

Example 2

50 Parts of ethylene/vinyl acetate copolymer (32—34% vinyl acetate) and 50 parts of a low density polyethylene (Alkathene WNC 18) were mixed on a two roll mill at 140° C. for 10 minutes, after which the mixture was sheeted off and moulded using a hot mould at 150° C. under a pressure of 210 Kg./cm.^{2} to give a sheet 0.14 cm. thick. "Alkathene" is a Registered Trade Mark.

Discs were cut from the sheet and inserted into shells as described in Example 1, except that the temperature of the first heating plate was raised to 100° C. The rest of the procedure was also as in Example 1. Similar carbonation retention tests were carried out on the products, and it was found that they gave good sealing performance.

Example 3

A sheet of the same ethylene/vinyl acetate copolymer as in Example 2 was moulded at 150° C at 210 Kg./cm.^{2} to give a sheet 0.14 cm. thick, from which liners were made as in Example 2. These liners were transparent, and were applied to lithographed vinyl coated crown. The lined crowns were tested in the same manner as those of Examples 1 and 2, and were found to have good sealing properties.

Example 4

A mixture of the following composition was prepared:

	parts
Polyvinyl Chloride (paste-making grade)	100
Dioctyl Phthalate	80
Azobiscarbonamide	3

The mixture was partially gelled as a thin sheet 0.18 cm. thick. Discs of diameter 1 cm. were cut from this sheet, and were inserted into internally lithographed crown shells internally coated with a vinyl lacquer. On heating and moulding as described in Example 1, gaskets were obtained in which the centre panel was clear whilst the raised annulus was opaque and had expanded to form a cellular cushion seal.

Example 5

A mixture of 50 parts of low density polyethylene as in Example 2, 50 parts of butyl rubber, isobutylene-isoprene copolymer "Polysar Butyl 101", having an unsaturation of 0.7 mole % and a Mooney Viscosity of 70.0 and 25 parts of the ethylene/vinyl acetate copolymer as used in Example 2 was formed into discs as described in that Example, and the discs incorporated in shells and cold-moulded as in Example 1, except that the base of the recessed holder 20 was curved to fit the shelf. The coating weight was 180 mg.

Each of Examples 1—4 could also be carried out using a recessed holder 20 curved to correspond to the curvature of the shell.

WHAT WE CLAIM IS:—

1. Process for the production of gasketed container closures which includes introducing a pre-formed insert of a solid thermoplastic resilient material into a container closure which is warmed sufficiently to cause adequate adhesion of the insert to the closure, and moulding the insert under pressure to the required shape with a cold-moulding member, if necessary after further heating the closure.

2. Process according to claim 1, wherein

the insertion and moulding of the thermoplastic material is carried out in essentially a single operation.

3. Process according to claim 1 or 2, wherein the container closure is a crown shell and the moulding member is of such a shape as to contour the thermoplastic material to give a relatively thick annular sealing portion and a thinner central panel which protects the metal or lacquer of the crown shell.

4. Process according to any preceding claim, which is carried out continuously.

5. Process according to any preceding claim, wherein the thermoplastic resilient material is one claimed in claims 1—24 of our Specification No. 33863/67 (Serial No 1112025).

6. Process according to any one of claims 1—4, wherein the thermoplastic resilient material is a composition claimed in our Specification No. 33862/67 (Serial No 1112024).

7. A process according to any one of claims 1—4, wherein the thermoplastic material is one of those specified in our Specification No. 1,092,161, plasticised polyvinyl chloride, or polyethylene, or a mixture of polyethylenes of different densities, molecular weights, or molecular weight distributions.

8. Process according to any preceding claim, wherein the thermoplastic resilient material comprises a blowing agent which is vaporised or decomposed on warming the insert, thereby forming a cellular gasket.

9. Process according to claim 1, substantially as herein described.

10. Process for the production of gasketed container closures substantially as herein described with reference to, or as illustrated in, Figures 8—13 of the accompanying Drawings.

11. Gasketed container closures obtained by a process claimed in any preceding claim.

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FIG. 1.

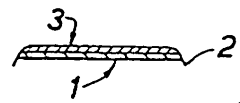


FIG. 2.

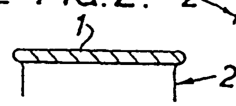


FIG. 3.

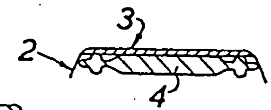


FIG. 4.

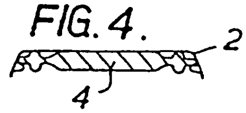


FIG. 5.

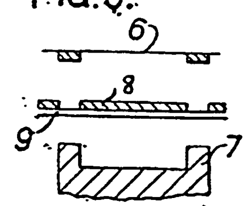


FIG. 6.

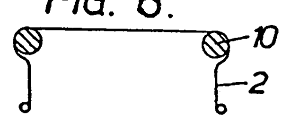
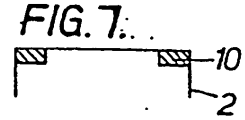


FIG. 7.



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Sheets 1 & 2

'IG. 3.

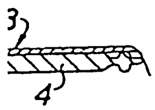


FIG. 8.

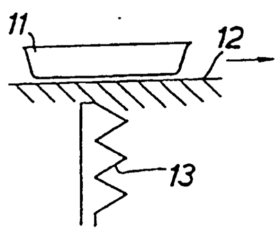


FIG. 9.

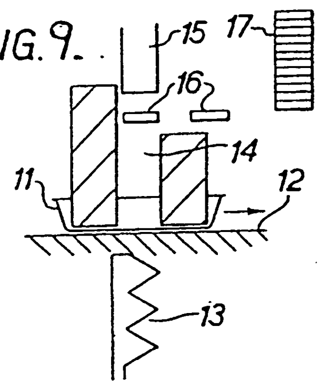


FIG. 10.

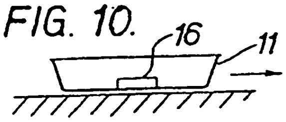
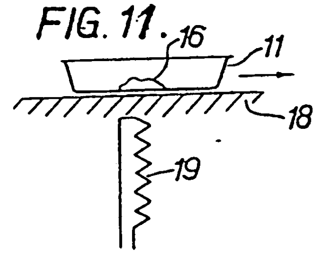


FIG. 11.



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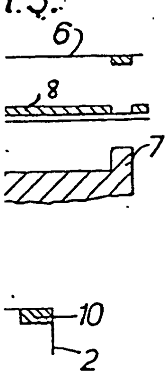


FIG. 12.

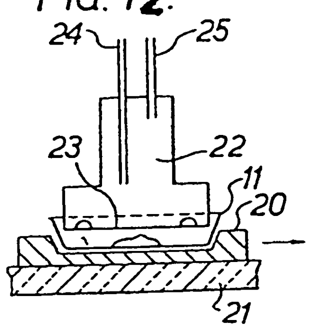
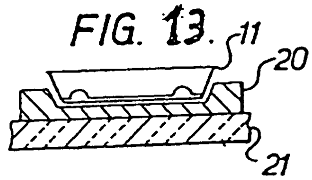


FIG. 13.



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Sheets 1 & 2

FIG. 1.

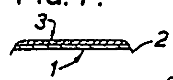

FIG. 2.

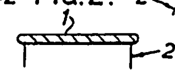

FIG. 3.

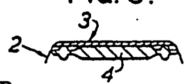

FIG. 4.

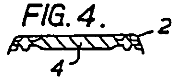

FIG. 5.

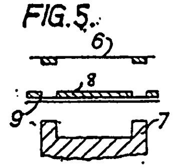

FIG. 6.

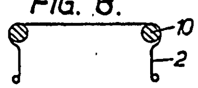

FIG. 7.

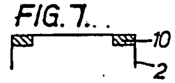

FIG. 8.

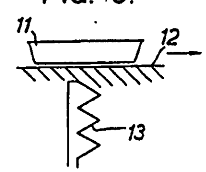

FIG. 9.

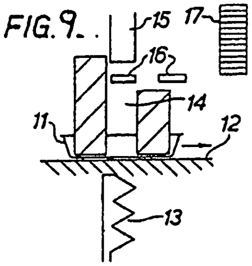

FIG. 10.

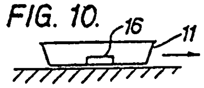

FIG. 11.

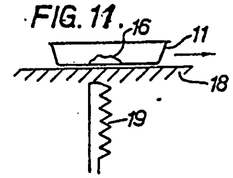

FIG. 12.

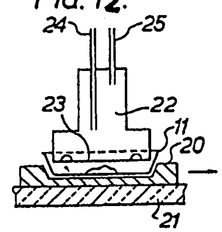

FIG. 13.